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Yttrium Oxide Nanowires

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1. Introduction

Yttrium is one of the most important rare-earth elements. Yttrium compounds show promise for applications such as high quality phosphors, catalysts, up-conversion materials and fine ceramics because of their optical, catalytic properties and high-temperature stability. Yttrium oxide is the most familiar yttrium compound, which is popularly known as host for ion doping of other rare earth elements. For example, europium doped yttrium oxide, Y₂O₃:Eu³⁺, is a superior red phosphor with a quantum efficiency of nearly 100%. It presents good luminescent characteristic, acceptable atmospheric stability, reduced degradation under applied voltages, and the lack of hazardous constituents as opposed to sulphide phosphors, thus it has commercial applications in fluorescent lamps, projection televisions, and field emission displays (FEDs). With respect to the physical properties, yttrium oxide has a high melting point ($T_m = 2430$ °C), which is higher than that of a number of other well-known oxides, such as alumina, zirconia, yttrium aluminum garnet (YAG), and spinel. Yttrium oxide exists as a cubic phase and is stable up to melting point without any phase transformations. Furthermore, it has a very large unit cell, which results in large unit slip distances. Hence, it is expected that plastic deformation in yttrium oxide by dislocation motion would be difficult. These properties endow it with usefulness as bulk ceramics for refractory applications. In addition, yttrium oxide has found applications in a wide variety of catalytic reactions owing to its basic nature. Therefore, the preparation of yttrium oxide, as well as its precursors, has attracted much academic attention. Yttrium oxide has been prepared in many shapes, like spherical particle (Tomaszewski et al., 1997; Sharma et al., 1998; Yang et al., 2007), rod (Wan et al., 2005), tube (Li et al., 2004; Fang et al., 2003; Tang et al., 2003; Wang et al., 2003; Zhang et al., 2008), prism (Zhang et al., 2005), plate (Wan et al., 2005) or sheet (Zhang et al., 2008), and wire. Our group has systematically investigated the phase distribution and morphology of products synthesized under hydrothermal conditions (Li & Yanagisawa, 2008). It was found that by simply adjusting the hydrothermal temperature and initial pH value of the starting solution, yttrium oxide with a diversity of well-defined morphologies like sheet, rod, needle and tube were successfully fabricated from different precursors. At the same time, the particle size of products could be controlled in a wide range.

Hitherto, the reported synthetic pathways to yttrium oxide nanowire were unlimitedly accomplished by the preparation of its precursors through hydrothermal reaction followed

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by decomposing these precursors into oxide. Hydrothermal reaction will not result in yttrium oxide directly, because yttrium oxide is not stable under hydrothermal conditions at temperature lower than 550 °C, according to the phase diagram of Y_2O_3 -H₂O system (Shafer & Roy, 1959).

Reactant	pН	Hydrothermal T.	Size	Reference
$Y_2O_3 + 10\%$ HNO ₃ + 10% N ₂ OH /KOH	13	180 °C for 12-24 h	Did not mention	Wang & Li, 2003
+ 10 % NaOT/ KOT				
$Y(NO_3)_3 \cdot 6H_2O + 10\%$	10	180 °C for 10 h	D: 30-40 nm;	Li et al., 2004
КОН			L: 1.4 µm	
Y_2O_3 + concentrated	12-13	170-180 °C for 12 h	D:100-300 nm;	Bai et al., 2005
HNO ₃			L: 1-2 µm	
+ 0.2M NaOH				
Y_2O_3 + concentrated	13	170 °C for 24 h	D: 85-95 nm;	Wu et al., 2005
HNO ₃			L: several tens of	
+ NaOH + PEG 2000			microns	
Y ₂ O ₃ + 3.0M HNO ₃	12	200 °C for 24 h	D: 40-100 nm;	Li & Yanagisawa,
+ ammoina solution			L: ~1.4 µm	2008
Y(NO3)3 6 H2O (5mol%	13	180 °C for 10 h	D: 50 ± 3 nm;	Zahir et al., 2009
Sm)			L: several tens of	
+15% TEAH			microns	

Table 1. Summarized literatures on $Y(OH)_3$ nanowires synthesized by precipitation-hydrothermal synthetic method

Wirelike yttrium hydroxide with hexagonal structure has been used as the precursor of yttrium oxide nanowires. Generally, the reported synthesis of yttrium hydroxide nanowires is accomplished through a two-step route, which is also called precipitation-hydrothermal synthetic method. Firstly, the precipitation of precursor colloids is obtained by adjusting the pH value of Y(NO₃)₃ aqueous solution, sometimes produced by dissolving Y₂O₃ in nitric acid, to 10-14 through the addition of NaOH/KOH solution. Then this precipitation is hydrothermally treated at 120-180 °C. The products are usually composed of discrete, singlecrystalline hydroxide nanowires with diameter of 30-300 nm. Corresponding oxide nanowires were received after thermal treating the hydroxide at 450-700 °C in air. For instance, Wang et al. reported the synthesis of hydroxide nanowires of yttrium and of other rare-earth elements through this synthetic method at 180 °C and pH around 13 (Wang & Li, 2003). The driving force for the growth was attributed to the crystal structure of yttrium hydroxide. Li et al. explained the formation mechanism of yttrium hydroxide nanowires by the complex interaction and balance between the chemical potential and the rate of ionic motion (Li et al., 2004). Europium doped yttrium oxide nanowires were also prepared by this method by mixing appropriate amount of europium oxide and yttrium oxide. The obtained Y₂O₃:Eu³⁺ nanowires exhibited strong red ⁵D₀-⁷F₂ transitions in fluorescent spectra. Our group found that using ammonia solution replacing NaOH/KOH as precipitation reagent, yttrium hydroxide nanowires could be obtained at pH 12.5 via hydrothermal reaction at 200 °C (Li & Yanagisawa, 2008). Several other researches also reported similar synthetic routes to yttrium hydroxide and yttrium oxide nanowires. Table 1 summarized the synthesis conditions and product information obtained in literatures. However, the hydroxide and oxide nanowires prepared through this synthetic method usually exhibit low

aspect ratio, at most 50, which makes it look more like needles than wires, as shown in Fig. 1. Yttrium hydroxide nanowires with higher aspect ratio could be prepared by a polymerassisted hydrothermal method (Wu et al., 2005). It is presumed that polyethylene glycol (PEG) played an important role on the formation of hydroxide nanowires. However, the final products coexisted with a mixture of nanorods and nanobelts. Recently, it is reported that after the doping of 5 mol% Sm³⁺ into yttrium and using tetraethylammonium hydroxide (TEAH) as precipitator, very well separated yttrium hydroxide nanowires with high aspect ratio were prepared (Zahir et al., 2009). The authors attributed the growth of yttrium hydroxide nanowires to the presence of Sm³⁺ and tetraethylammonium ion.



Fig. 1. TEM image of hexagonal $Y(OH)_3$ needles prepared by precipitation-hydrothermal synthetic method. The reaction was conducted at pH 12.5, 160 °C for 24h, with ammonia solution as precipitator

Our group has recently exploited a simpler one-step hydrothermal synthetic method to prepare yttrium hydroxide nanowires, where yttrium oxide powder instead of soluble yttrium salts was directly used as a starting material, and the reaction was conducted under near neutral conditions. Unlike yttrium hydroxide nanowires obtained by precipitation-hydrothermal synthetic method, the nanowires synthesized via this one-step method showed unusual, bundle-like morphology with much higher aspect ratio (Li et al., 2009). Furthermore, we found that two other yttrium compounds, yttrium oxide nitrate hydroxide (Li & Yanagisawa, 2008) and yttrium chloride hydroxide could also be prepared in wire shape in nanometer size. Both of them were synthesized through precipitation-hydrothermal synthetic method, using yttrium nitrate and yttrium chloride as starting material respectively. In this chapter, we will introduce the preparation and characterization of the three wirelike yttrium compounds, as well as their conversion to yttrium oxide nanowires.

2. Yttrium oxide nanowires prepared from yttrium hydroxide

As has introduced above, the yttrium hydroxide nanowires prepared from the precipitationhydrothermal synthetic method usually exhibit low aspect ratio. Recently, we synthesized

yttrium hydroxide nanowires by an acetic acid (HAc) assisted hydrothermal method under near neutral conditions. The as-synthesized nanowires are of higher aspect ratio up to 2000. The preparation was effected through a one-step hydrothermal procedure, simpler than the precipitation-hydrothermal synthetic method. Typically, the appropriate quantities of Y₂O₃ powder and 0.067 mol/L acetic acid aqueous solution were mixed at room temperature and hydrothermally treated at 200 °C for 24 h under agitation. Powder X-ray diffraction (XRD) characterization indicated that the product could be indexed to a pure hexagonal phase of Y(OH)₃, with lattice cell constants of a = 6.2610Å and c = 3.5440Å, identical to the reported data in JCPDS cards (JCPDS 83-2042), as shown in Fig. 2a. The (110) reflection around 2θ of 28° is much more pronounced compared with the literature data, indicating the preferred growth direction of these nanowires is along *c*-axis. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) observations indicated that the product was composed of bundles of nanowires, as shown in Fig. 3a-c. These bundles were relatively uniform in size with length of 30-40 µm. Each bundles consisted of numerous of nanowires with diameter of 20-50 nm. Assuming each nanowire spans the whole length of the bundle, the nanowires had an aspect ratio of 600-2000, which was much higher than that of the nanowires fabricated from the precipitation-hydrothermal synthetic method. High resolution transmission electron microscopic (HRTEM) image and selective area electron diffraction (SAED) pattern taken from an individual nanowire revealed its single-crystalline nature, as shown in Fig. 3d, e. After calcinations at 500 °C, the obtained yttrium oxide nanowires almost remained unchanged in morphology except that the wire diameter decreased slightly, owing to the higher density of oxide compared with that of hydroxide, as shown in Fig. 3f. The decomposition behaviours of yttrium hydroxide and other precursors will be summarized later.



Fig. 2. Typical XRD patterns of three precursors of yttrium oxide. (a) hexagonal Y(OH)₃, (b) $Y_4O(OH)_9(NO_3)$ and (c) yttrium chloride hydroxide hydrate

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Fig. 3. SEM (a, b), TEM (c), HRTEM (d) and SAED (e) images of bundles of $Y(OH)_3$ nanowires. SAED pattern was taken from the region indicated by white circle in (c). (f) TEM image of corresponding Y_2O_3 nanowires.

It was found that the formation of yttrium hydroxide nanowires was closely correlated with the presence of HAc and its concentration. Hydrothermal reaction conducted in pure water suggested that a majority of yttrium oxide could be converted into hydroxide after 72 hours of hydrothermal treatment, indicating that HAc was not necessary for this transformation. This result was consistent with the Y_2O_3 -H₂O phase diagram. However, the conversion process without the participation of HAc was very slow, and the product morphology totally differed from that of yttrium hydroxide prepared with the assistance of HAc. Our experiment results proved that in the presence of HAc the whole reaction was completed within several hours. Moreover, it was also found that the product morphology was sensitive to HAc concentration. Yttrium hydroxide could be prepared at concentration between 0.001 and 0.01 mol/L. The optimum HAc concentration for the growth of uniform nanowires was 0.006-0.007 mol/L. Lower concentration would result in irregular rods in a wide scale of size, while higher concentration would lead to the formation of an unknown phase, probably being yttrium acetate compound.

The investigation on the intermediate products obtained during different growth stages suggested that with the process of time, yttrium oxide disappeared step by step, while the appearance of hydroxide was observed at the same time. The transformation from oxide into hydroxide was completed within two hours. SEM images (Fig. 4) indicated that at the early stage of hydrothermal reaction, the sample was composed of irregular blocks and a small quantity of microrods with quasi-hexagonal geometry, which was unreacted yttrium oxide and the formed hydroxide, respectively. The individual microrod was constructed by numerous of nanorods fused in the same orientation. Along with the proceeding of hydrothermal reaction, these rods became less faceted, and the diameter is not uniform along the long axis, forming a spindle-like structure. The tips of the rod looked like tubes. As the reaction proceeded, the size of these microrods, as well as the aspect ratio increased, suggesting that the growth along the long-axis was faster than that along the short-axis. This result was supported by XRD results that the intensity of (110) diffraction of yttrium hydroxide increased remarkably in comparison with that of (101) diffraction. Subsequently, the tips of these rods began to split into wires and the whole rod developed into a bundle of nanowires eventually.

It is likely that the growth is accomplished through an oriented attachment and the subsequent dissolution process from the defect sites. Firstly, yttrium oxide gradually dissolves into the solution, and yttrium hydroxide nucleates into 1D nanocrystals owing to the high anisotropic structure along *c*-axis of hexagonal phase. These nanocrystals then align side by side and fuse together spontaneously by oriented attachment (Penn & Banfield, 1999; Banfield et al., 2000; Penn et al., 2001), giving rise to the formation of quasi-single crystals, where the thermodynamic driving force is the substantial reduction of surface energy contributed by the interface elimination. During that procedure, some defects like dislocation are introduced into the microrods as a result of imperfect attachment (Penn & Banfield, 1998), which accumulates during the crystal growth. Therefore, the microrod gradually loses its anisotropy. When the starting yttrium oxide is depleted, the reactant concentration in the solution begins to drop. As the concentration drops to a certain level, the particles stop its growth on the whole. However, because the site where defect exists has a higher solubility than those free of defects, there is an internal concentration gradient. Consequently, the microrods dissolve from the defects and grow onto other facets to maintain the solubility equilibrium, resulting in bundles of nanowires. This process is

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similar as the intraparticle ripening mechanism proposed by Peng et al (Peng & Peng, 2001). The formation mechanism of the nanowires is schematically shown in Fig. 5. The growth of the yttrium hydroxide nanowires begins from 1D nanocrystals, which align in the same direction and fuse together via oriented attachment process, leading to microrods. These microrods grow into tube-like structures by loss of anisotropy and growing preferable along circumference. Finally, the microrods are dissolved from the defect sites and split into nanowires.



Fig. 4. SEM images of products synthesized at 200 °C in 0.067 mol/L HAc for 0h (a, b), 0.5h (c, d), 1.0 h (e, f) and 2.0 h (g, h)

HAc plays a key role in the formation of the unusual nanowires, probably acting in two ways. Firstly, the addition of HAc changes the pH value of solution, thus accelerates the conversion from yttrium oxide to hydroxide. Secondly, acetic acid molecules or ions adsorb to the surface of yttrium hydroxide crystallites, introducing defects and blocking the attachment mechanism.



Fig. 5. Sketch for the growth mechanism of Y(OH)₃ nanowires. Reprinted with permission from Cryst. Growth Des, 9 (2):978-981, Copyright 2009 American Chemical Society.

3. Yttrium oxide nanowires prepared from yttrium oxide nitrate hydroxide

As we have mentioned in the introduction of this chapter, yttrium hydroxide nanowires could be prepared through a precipitation-hydrothermal synthetic method. Our group have systematically investigated the precipitation-hydrothermal reaction conducted in a wide range of temperature and pH value. It was found that three compounds, hydrated yttrium nitrate hydroxide $Y_2(OH)_{5.14}(NO_3)_{0.86}$, yttrium oxide nitrate hydroxide $Y_4O(OH)_9(NO_3)$, and hexagonal yttrium hydroxide $Y(OH)_3$ were obtained when hydrothermal reaction were conducted at 80-220 °C and pH 6.0-13.5. $Y_2(OH)_{5.14}(NO_3)_{0.86}$ and $Y_4O(OH)_9(NO_3)$ occurred under near neutral to weak basic conditions, and low hydrothermal temperature would result in $Y_2(OH)_{5.14}(NO_3)_{0.86}$, while $Y_4O(OH)_9(NO_3)$ was received at higher temperature. If the hydrothermal reaction was conducted under strong basic condition, $Y(OH)_3$ was obtained. The pH boundary where $Y(OH)_3$ was formed was 11.25 and 13.0 when ammonia and NaOH solution was used as precipitator respectively. Among the three yttrium compounds, yttrium oxide nitrate hydroxide $Y_4O(OH)_9(NO_3)$ could also be synthesized in wire shape, which made it a alternative precursor for the preparation of yttrium oxide nanowires.

The synthesis of $Y_4O(OH)_9(NO_3)$ was almost identical with that of yttrium hydroxide with the exception that the reaction was conducted at a slightly lower pH value. In a typical synthesis process, an appropriate quantity of yttrium oxide was dissolved in nitric acid. Then ammonia/NaOH solution was added to adjust the solution to a designated pH value. Products were received after hydrothermal treatment at 80-220 °C for 24 h and then investigated by XRD and TEM characterizations. As the XRD pattern shown in Fig. 2b, the product could be attributed to a pure monoclinic phase. TEM images (Fig. 6a, b) illustrated that these nanowires were straight and well-dispersed with uniform diameter along the longitudinal axis. The diameter ranged from 30 to 50 nm, while the length was up to tens of micrometers. Some nanowires were as long as several millimetres, spanning the whole visual field under the TEM observation at low magnification (×4). That means the nanowires could be prepared with extraordinary high aspect ratio. Some of the shorter nanowires probably were broken fragments from the longer ones by the ultrasonic during the TEM pre-treatment. HRTEM and SAED characterization revealed its single crystalline nature. It seems that the driving force for the spontaneous growth of nanowires is the anisotropic characteristic of monoclinic structure of yttrium oxide nitrate hydroxide. The yttrium oxide obtained through calcinations maintained the wire shape. However, it became porous in contrast with its precursor, as shown in Fig. 6c.



Fig. 6. TEM images of $Y_4O(OH)_9(NO_3)$ nanowires hydrothermally synthesized at 200 °C, pH 12.5 (a, b), and of Y_2O_3 nanowires prepared thereby (c)

This yttrium compound could be prepared in a wide pH range, from 6.0 to 13.0. However, nanometer sized product was only obtained at relatively high pH value, for example, pH 12.5, while low pH value would lead to the formation of microrods with hexagonal cross-section. Along with the increasing pH value, the size of the product decreased. This is because that high pH value leads to the formation of large amount of nuclei and small particles is favoured due to the suppressed growth of each nuclear. It should be noted that further increasing the pH value would give rise to the formation of yttrium hydroxide, which contains more hydroxyl in its structure and thus is more stable than yttrium oxide nitrate hydroxide under stronger basic conditions. Therefore, by carefully controlling the pH value of the colloidal solution and its concentration, nanosized products could be prepared. The effects of hydrothermal temperature on products were also studied by fixing the pH

value and changing the reaction temperature from 140 °C to 220 °C. The products did not show much difference in morphology except for the size distribution narrowed down with increasing temperature, indicating that pH value was the predominant factor in strong basic media, and the morphologies were less dependent on reaction temperature as compared with pH value.

4. Yttrium oxide nanowires prepared from yttrium chloride hydroxide

In the aforementioned precipitation-hydrothermal synthesis, it is yttrium nitrate that was used as starting material. What will happen if yttrium chloride took the place of yttrium nitrate? Our study revealed that different products were received in this case, and Cl element was usually involved in the final products. Among them, an yttrium chloride hydroxide with indefinite composition could be prepared in wire shape.



Fig. 7. SEM image of yttrium chloride hydroxide nanowires

Similar as that of $Y_4O(OH)_9(NO_3)$, the synthesis was started by dissolving yttrium oxide in hydrochloric acid. Ammonia solution was then added to adjust the solution to designated pH value. Flocculent product was received after hydrothermal treatment at 200 °C for 12 h. The crystal structure and morphology of the products were studied by XRD and SEM, respectively. Fig. 2c shows the XRD pattern of the product hydrothermally synthesized at pH 9.5. It was poorly crystallized and could not be identified to any known phase. SEM observation revealed that the product consisted of sub-micrometer wires. The wires were of uniform diameter ranging from 100 to 300 nm, while the length was up to tens of microns, as shown in Fig. 7. It is possible that the wirelike product synthesized at pH 9.5 belongs to chloride hydroxide hydrate, represented by $Y(OH)_xCl_{3-x}$ yH₂O. These wirelike products could be prepared within the pH range from 9.50 to 10.25.

5. Decomposition behaviours of yttrium oxide precursors

All of the three above-mentioned yttrium compounds could be converted into yttrium oxide through calcination in air. Their wirelike morphologies were maintained except for the

slight shrinkage in size, arising from the higher density of yttrium oxide compared with their precursors. Because of the difference in structural compositions, these compounds exhibited different characteristics during calcination. Their decomposition behaviours were illustrated in Fig. 8.



Fig. 8. Typical TG-DTA curves of (a) hexagonal Y(OH)₃, (b) Y₄O(OH)₉(NO₃) and (c) yttrium chloride hydroxide hydrate. Reproduced in part with permission from J. Solid State Chem. 181 (8):1738-1743, Copyright 2008 Elsevier.

During heat treatment, yttrium hydroxide nanowires synthesized by the HAc-assisted hydrothermal method underwent two step-wised decomposition procedures, where yttrium hydroxide was firstly transformed into an intermediate oxyhydroxide, YOOH, and then it converted into oxide at elevated temperature. The conversions occurred at around 280 and 415 °C, respectively, as indicated by two DTA endothermic peaks in Fig. 8a.

For Y₄O(OH)₉(NO₃) nanowires, there were two decomposition procedures during the heat treatment, as shown in Fig. 8b. Ion chromatography analysis suggested that the NO₃content of intermediate products did not decrease until temperature increased to 460 °C, indicating that N-O species were released in the second step. The weight loss of the first procedure between 330 °C to 450 °C was 10.14%, lower than the theoretical value associated

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with the total release of water (13.8%). It suggests that some hydroxyl was stilled remained in the structure. Following this step, sample transformed into oxide at around 490°C, accompanied by the release of N-O species and water.

With respect to yttrium chloride hydroxide, it also underwent two decomposition procedures during calcinations, as shown in Fig. 8c. The first one below 400 °C gave weight loss of 14.0%, which was consistent with the release of water, and the weight loss of 9.2% during the second step corresponded to the release of HCl. Its calcination behaviour exhibited an overall weight loss of 23.2%.

6. Conclusion

In summary, we have prepared three wirelike yttrium compounds by hydrothermal method, which were hexagonal yttrium hydroxide, yttrium oxide nitrate hydroxide, and yttrium chloride hydroxide. Hexagonal yttrium hydroxide nanowires were synthesized as single crystals from yttrium oxide powder by a simple acetic acid-assisted hydrothermal method under near neutral conditions. The nanowires show unusual, bundle-like morphology with diameter of 20-50 nm and aspect ratio of 600-2000. The growth of nanowires involved the oriented attachment of 1D nanocrystals to form microrods and selective dissolution from defect sites to form bundles of nanowires. Another wirelike yttrium compound, $Y_4O(OH)_9(NO_3)$ were obtained through conventional precipitation-hydrothermal synthetic route under basic condition. These nanowires were straight and well-dispersed single crystals, with extraordinary high aspect ratio and uniform diameter ranging from 30 to 50 nm. By substituting yttrium nitrate with yttrium chloride, yttrium chloride hydroxide nanowires with diameter of 100-300 nm and length up to tens of microns were synthesized at pH 9.50-10.25 via hydrothermal reaction.

All of the three compounds could be utilized to fabricate yttrium oxide nanowires through thermal treatment. This one dimensional (1D) structure may lead to new opportunities in yttrium chemistry. For example, the yttrium oxide nanowires showed intriguing high-temperature stability. Our experiments suggested that after calcination at 1400 °C, the yttrium oxide nanowires obtained from yttrium chloride hydroxide remained their wire shape, which makes it promising material for refractory applications, such as refractory insulation and high temperature gas filtration. Furthermore, the formation of the three compounds all went through a dissolution-crystallization procedure. Therefore, homogeneous rare earth doped yttrium oxide as starting material. By this means, 1D luminescent materials like Y_2O_3 :Eu³⁺ nanowires can be fabricated, which may extend its application and benefit the understanding of the luminescent mechanism in low dimensional materials.

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This book describes nanowires fabrication and their potential applications, both as standing alone or complementing carbon nanotubes and polymers. Understanding the design and working principles of nanowires described here, requires a multidisciplinary background of physics, chemistry, materials science, electrical and optoelectronics engineering, bioengineering, etc. This book is organized in eighteen chapters. In the first chapters, some considerations concerning the preparation of metallic and semiconductor nanowires are presented. Then, combinations of nanowires and carbon nanotubes are described and their properties connected with possible applications. After that, some polymer nanowires single or complementing metallic nanowires are reported. A new family of nanowires, the photoferroelectric ones, is presented in connection with their possible applications in non-volatile memory devices. Finally, some applications of nanowires in Magnetic Resonance Imaging, photoluminescence, light sensing and field-effect transistors are described. The book offers new insights, solutions and ideas for the design of efficient nanowires and applications. While not pretending to be comprehensive, its wide coverage might be appropriate not only for researchers but also for experienced technical professionals.

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